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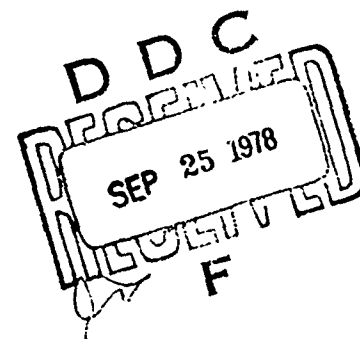


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EVALUATION OF SUPERHARD TRANSPARENT COATINGS

JOHN R. PLUMER
COMPOSITES DIVISION

February 1978



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ARMY MATERIALS AND MECHANICS RESEARCH CENTER
Watertown, Massachusetts 02172

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ABSTRACT

A test program was carried out to assess hard surface coatings developed for reducing scratching problems encountered with plastic glazings used in Army aircraft.

The tests employed simulated aircraft flight and handling conditions, and consisted of the following: a windshield wiper test, a felt-pad abrader test, solvent resistance, hardness, impact, ultraviolet exposure, humidity, and weatherometer testing.

The coatings were found to substantially increase the abrasion resistance of several types of plastic materials; water and ultraviolet radiation were factors adversely affecting coating performance.

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INTRODUCTION

The majority of Army helicopters currently use an acrylic plastic as a glazing material; however, recent combat service has demonstrated these windshields are extremely prone to scratching (due to wiper blade action and prop-wash blown dust) and require frequent replacement. Acrylic glazing also produces potentially dangerous spall on foreign object impact (e.g., blown rocks, small arms fire). Previous work¹ has shown that polycarbonate plastic, coated with a protective hard-surface material, offers a solution to spallation, while the hard surface coating significantly increases the resistance to surface-produced abrasion. The feasibility of coated polycarbonate UH-1 helicopter windshields has been demonstrated through flight testing. Typical available coatings, however, afford protection for a limited time as most are susceptible to extremes of sunlight and humidity.

The objective of this test program is to evaluate the performance, in a simulated windshield regime, of a unique concept of hard surface coating developed for transparent plastic. The "Super Hard Transparent Coatings" were developed and applied to specimens used in this effort by Marks Polarized Corporation, Whitestone, New York, under Eustis Directorate, USAAMRDL Contract DAAJ02-73-C-0062. This contract represents part of a concerted effort by AVRADCOM to upgrade the performance and reliability of transparencies on all classes of Army aircraft. The testing phase of the program was established with AMMRC specifically to duplicate the tests that were described in the contractor's test plan in order to substantiate and verify independently the contractor's test results. Additional tests were performed to complete the evaluation of the candidate materials, Super Hard Transparent Coatings, for possible use in Army aircraft windshields.

MATERIALS

The coatings under investigation, 129c (70% silica, 30% polyvinyl alcohol) and 130 (15% aluminum oxide, 70% silica, 15% polyvinyl alcohol), were applied to the following plastics: polycarbonate, Plex II, and stretched acrylic. These coatings employ a transparent polymer (or coating) containing hard submicron particles held in suspension. The submicron particles do not appreciably scatter light because of their small size (diameter of approximately 100 Å).

TESTING INSTRUMENTATION AND PROCEDURE

Abrasion Resistance

The ability of a transparent polymer to withstand mechanical and nonmechanical abrasion, with a minimal loss of optical properties, is of fundamental interest. Abrasion resistance of a transparent polymer may then be determined

1. PLUMER, J. R. *Development of Scratch and Spall-Resistant Windshields*. Army Materials and Mechanics Research Center, AMMRC TR 74-19, August 1974.

by quantitatively measuring the haze produced by a given test method. For materials such as polycarbonate, acrylic, and stretched acrylic, the scratch resistance of coatings is a necessary determination when considering aircraft applications.

The problem of evaluating plastics and coatings for abrasion resistance was approached by utilizing a test apparatus designed to simulate field conditions. A windshield wiper test apparatus was used to approximate in-flight effects on windshields.¹ The apparatus shown in Figure 1 allowed control of such variables as wiper speed, abrasive, arm pressure, and monitoring of the test conditions.

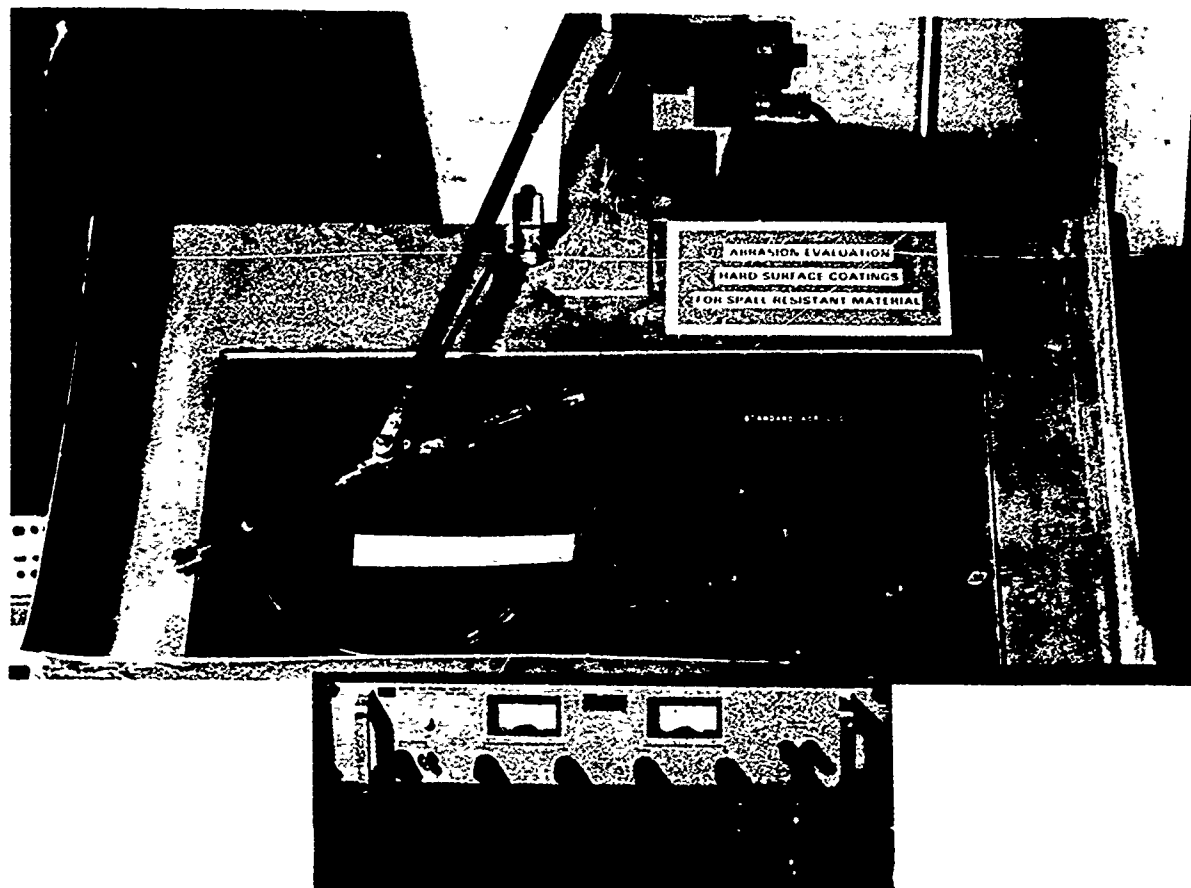


Figure 1. Windshield wiper apparatus.
19-066-632/AMC-76

Windshield Wiper Test

All samples were cut to a size of 6" x 6" and flush fitted into a mask for mounting. The abrasive slurry used for all testing consisted of A.C. air cleaner test dust, coarse (50% 30 to 80 micron) size, continuously suspended in solution by a mechanical stirrer. The slurry contains 300 grams of grit per three liters of water. Flow rate was regulated at approximately 300 ml per minute and

discharged between samples. Cycle speed was maintained at 100 cycles per minute, the approximate low speed of a UH-1 helicopter, while wiper load was fixed at six pounds. After every 1000 cycles samples and control were removed from the testing table, rinsed and dried, and evaluated for light and haze transmission as described in ASTM D1003-61. A Hunter Lab color-color difference meter (model D250P2) was used for light conversion and haze determination throughout the test program. Results are listed in Table 1.

Table 1. ABRASION RESISTANCE WINDSHIELD WIPER TEST

Cycles	Polycarbonate						Plex II						Stretched Acrylic					
	Uncoated		129c		130		Uncoated		129c		130		Uncoated		129c		130	
	%T	%H	%T	%H	%T	%H	%T	%H	%T	%H	%T	%H	%T	%H	%T	%H	%T	%H
0	87.7	1.2	89.4	1.2	89.4	1.2	93.8	0.70	93.2	0.64	93.2	0.64	97.0	0.80	93.6	0.84	93.1	1.1
500	84.0	15.3	89.2	3.8	89.0	2.2	91.5	20.0	93.3	0.93	92.6	2.1	92.5	18.5	92.9	1.1	93.0	1.7
1,000	84.0	21.0	89.2	5.8	89.0	2.8	89.6	23.5	93.0	1.2	91.3	2.7	90.5	21.5	92.2	1.9	92.9	2.1
2,000	-	-	89.0	6.0	88.6	3.3	-	-	93.2	1.2	91.2	2.6	-	-	91.9	1.9	92.9	2.1
3,000	-	-	89.0	6.1	88.0	5.3	-	-	93.7	1.2	91.1	2.6	-	-	91.2	2.2	91.9	3.1
4,000	-	-	89.0	6.1	88.4	5.1	-	-	91.5	1.9	91.1	2.5	-	-	91.0	2.4	91.5	3.5
5,000	-	-	89.0	6.4	88.3	5.6	-	-	91.3	2.2	89.8	2.5	-	-	90.6	2.5	91.5	3.5
6,000	-	-	88.9	6.7	88	6.4	-	-	91.0	2.4	89.5	2.9	-	-	90.4	2.8	91.2	3.8
7,000	-	-	88.7	7.5	88.3	6.7	-	-	90.9	2.3	89.5	3.0	-	-	90.1	2.9	91.0	3.9
8,000	-	-	88.6	7.0	87.7	6.6	-	-	90.5	2.5	89.2	3.3	-	-	89.8	3.9	89.8	4.0
9,000	-	-	87.9	7.3	87	6.7	-	-	90.7	2.2	89.0	3.2	-	-	89.8	3.0	89.7	4.4
10,000	-	-	87.8	7.8	87	6.8	-	-	90.6	2.5	89.0	3.7	-	-	88.1	2.2	89.1	4.5

All values represent an average of 3 test runs per sample
 Temperature at time of testing 72 F, 35% RH
 %T = % Transmission
 %H = % Haze

Reciprocating Arm Felt Pad Abrader

Evaluation of abrasion effects were also made by using a reciprocating arm felt pad abrader, designed to approximate conditions encountered by field cleaning of helicopter windshields by aircraft personnel, Figure 2. This unit provides an approximation of the nonmechanical cleaning action, such as hand wiping by the crew, of a windshield during operation of the helicopter. This unit is preferred over the Taber abrader because the abrasive area is larger, 1" x 2", and is designed to fit the inspection area exactly in the Hunter Lab Optical Head.

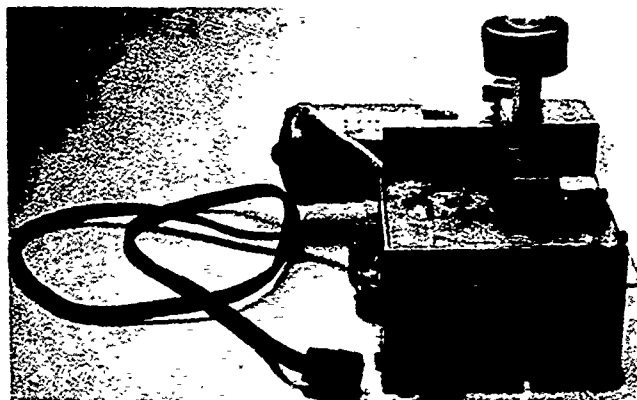


Figure 2. Reciprocating arm felt pad abrader.
 19-066-631/AMC-76

All samples were cut to a uniform size, 6" x 6", permitting the samples to be precisely refitted into the optical head and the abrader unit. A one-inch-diameter disk, 100% wool felt 1/8" thick, was cemented to the abrader head. The reciprocating speed was maintained at 50 cycles per minute; a 500-gram load was applied to the head during testing. Samples were placed into the unit with their coated side facing the abrader head. The felt pad was impregnated with 60 grit Norbide boron carbide abrasive and placed in the unit, this being repeated after every 100 cycles. Samples were removed after every 100 cycles and all abrasive material was removed from the evaluating area. Optical properties were then measured. Tests were continued until 10% haze or 1000 cycles were reached. Results are given in Table 2.

Table 2. ABRASION RESISTANCE, FELT PAD RECIPROCATING ARM ABRADER

Cycles	Polycarbonate			Plex II			Stretched Acrylic		
	Uncoated	129c	130	Uncoated	129c	130	Uncoated	129c	130
0	2.8	1.3	1.6	1.3	1.1	1.8	1.7	1.7	1.6
1	9.9	-	-	-	-	-	-	-	-
2	13.1	-	-	-	-	-	-	-	-
3	16.7	-	-	-	-	-	-	-	-
4	22.1	-	-	-	-	-	-	-	-
5	23.1	-	-	12.9	-	-	12.8	-	-
6	28.7	-	-	-	-	-	-	-	-
10	-	-	-	17.1	-	-	17.1	-	-
15	-	-	-	19.5	-	-	22.0	-	-
20	-	-	-	25.2	-	-	27.0	-	-
100	-	1.7	2.3	-	1.4	2.2	-	1.8	2.1
200	-	1.9	3.1	-	1.5	2.6	-	2.2	2.7
300	-	2.0	3.5	-	2.7	3.0	-	2.0	3.2
400	-	2.8	3.7	-	2.4	3.4	-	2.1	3.3
500	-	4.0	3.9	-	2.5	4.2	-	2.2	3.3
600	-	4.4	4.6	-	2.7	3.8	-	2.4	3.6
700	-	4.2	5.9	-	2.9	4.6	-	2.6	3.8
800	-	4.8	5.9	-	3.0	4.7	-	2.8	4.1
900	-	5.1	6.3	-	3.4	5.5	-	2.9	4.2
1,000	-	5.4	7.5	-	3.5	5.6	-	3.4	4.2

All values are given in percent haze, ASTM 1000-3

Solvent Resistance

The coated side of each transparent polymer was subjected to 15 minutes of exposure to the following solvents:

Methyl Ethyl Ketone	Toluene	Methyl Alcohol
Methyl Isobutyl Ketone	Hexane	Butyl Alcohol
Acetone	Xylene	Isopropyl Alcohol

An eyedropper was used to aid in dispensing solvents uniformly. After exposure they were rinsed and dried, and their optical properties, light transmission and haze transmission, were measured. Results are listed in Table 3.

Mohs' Hardness

The Mohs' hardness scale was used to assess hardness of the coated plastic samples. The scale determines hardness by comparison of test materials with natural minerals. For a given Mohs' hardness, rubbing two materials any number of

Table 3. SOLVENT TESTING* OF SUPERHARD TRANSPARENT COATINGS

Solvents	Polycarbonate			Plex II		
	Uncoated	129c, %	130, %	Uncoated	129c, %	130, %
None	1.3%	1.3	1.5	0.7%	0.7	1.3
Hexane	No Effect	1.6	1.6	No Effect	1.4	2.0
Xylene	Severe Blistering†	2.2 Crazing	14.4 Severe Crazing	Slight Crazing	0.85	1.6
Toluene	Severe Blistering†	4.7 Crazing	32.4 Severe Blistering	Slight Crazing	0.85	2.0
Acetone	Blistering	4.0 Slight Crazing	10.4	Clouding	0.75	2.6
Butyl Alcohol	No Effect	1.6	1.5	No Effect	0.1	2.2
Isopropyl Alcohol	No Effect	3.6	3.0	No Effect	1.7	3.6
Methyl Alcohol	No Effect	1.8	1.5	Slight Crazing	0.85	1.8
Methyl Isobutyl Ketone	Ground Glass Look Effect†	3.5	3.4	Slight Crazing	0.9	1.3
Methyl Ethyl Ketone	Severe Blistering	2.2	10.4 Severe Crazing	Ground Glass Look Effect†	0.9	2.0

*All numerical data is represented as percent haze. All optical measurements were conducted with the coated side of the sample facing the light source.

†Some tests produced haze too extreme to accurately determine.

cycles or at any pressure will not produce visible scratching if the Mohs' hardness of the material exceeds that of the abrasive (mineral). All tests were conducted on the coated side of the sample. The results show that all samples have a Mohs' hardness of approximately 3.5 to 4.0. The scale used consisted of the following minerals (the number identifies the Mohs' hardness value): 1. Talc; 2. Gypsum; 3. Calcite; 4. Fluorite; 5. Apatite; 6. Feldspar; 7. Quartz; 8. Beryl.

Impact Testing

Impact testing was conducted in accordance with the method outlined in the contractor's test plan. A 200-gram steel ball was dropped from a height of 50 cm, utilizing a Visotek Model FD 9150 impact test apparatus, after which the sample was evaluated for any apparent fracture or separation of the coating from the substrates. Samples of 129c and 130 coatings on Plex II, polycarbonate, and stretched acrylic were tested; none of the samples suffered any apparent fractures or separation of the coatings from the substrate.

Ultraviolet Testing

A temperature-controlled aging chamber equipped with an ozone-generating ultraviolet lamp (8 watts over a range of 2537 Å through 4360 Å) was used for the test. Samples were positioned with the coated side toward the lamp and six inches from the source. Chamber temperature was maintained at 75 C. Samples

were removed and inspected for crazing and haze development on an hourly basis for the first eight hours of the test cycle, and every 24 hours for the full duration of the 120-hour test. In general, this accelerated regime is about seven times more severe than outdoor exposure at a hot-dry site. This test method is based on procedures outlined in MIL-STD-810-D. The results of exposure and abrasion testing are shown in Table 4.

Table 4. ULTRAVIOLET EXPOSURE AND ABRASION TESTING

Ultraviolet Radiation Effect	Exposure		Uncoated		Stretched Acrylic	Plex II		PC*		
	Day	Hour	Plex II	PC	129c	129c	130	129c		
		1	No Change	No Change	No Change	No Change	No Change	No Change		
		2			No Change					
		3			No Change					
	1	4			Cracking					
		5								
		6								
		7								
		8					Cracking	Cracking		
	2	32								
	3	56								
	4	80								
	5	104								
	6	120	↓	↓	↓	↓	↓	↓		
Post Exposure, Felt Pad										
Abrasion Test	Cycles		%T	%T	%T	%H	%T	%H	%T	%H
	0		93.0	88.7	89.7	2.00	93.0	1.39	89.3	2.25
	200		Values Unchanged from Table 1		89.6	2.56	92.5	1.29	88.6	3.0
	400				90.3	2.76	92.0	1.84	87.1	3.8
	600				89.8	3.00	91.0	2.08	87.1	4.0
	800				89.7	3.34	90.0	3.22	87.6	4.6
	1000						90.5	3.20	86.0	5.9
	1200						90.2	3.43	81.2	6.0

*Polycarbonate

Water Immersion Test

Samples were submerged in distilled water maintained at 70 F; periodic removal, air drying, and inspection was made to determine any development of micro-cracking, haze, or deterioration of the coating surface. A felt-pad abrasion test was conducted at the conclusion of the 48-hour immersion test. Some results of the testing are shown in Table 5. Polycarbonate coated with 130 showed both a very rapid development of microcrazing (less than one hour) and a significant reduction in abrasion resistance. Twice as much haze developed on the water-soaked samples as was produced by similar testing on virgin coated (130) polycarbonate.

Cracking developed on the 129c polycarbonate after 16 hours of water immersion; however, abrasion resistance was decreased only slightly. The optical properties of all samples appeared unchanged by the water immersion. No significant change was noted in abrasion properties on the Plex II coated samples.

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Table 5. WATER IMMERSION (70 F) AND ABRASION TESTING

Hours of Immersion	Polycarbonate		Plex II	
	129c	130	129c	130
1	Unchanged	Microcracking	Unchanged	Unchanged
2				
3				
4				
8				
16	Microcracking			
24				
32				
40				
48				

Felt Pad Abrasion Tests, 48 hr of Water Immersion

Cycles	%T	%H	%T	%H	%T	%H	%T	%H
0	89.0	3.2	88.8	2.7	89.3	2.3		
200	82.8	7.0	85.9	4.3	89.9	3.3	81.5	7.6
400	82.4	5.75	85.2	4.8	87.2	4.5	80.4	10.7
600	78.0	9.9	84.6	6.0	86.4	5.4	85.1	12.1
800	74.0	14.4	83.0	7.9	85.5	7.2	83.4	14.8
1000	66.8	14.9	86.9	12.0	85.0	7.5	83.3	15.8

Weatherometer Evaluation

Weatherometer testing was conducted using an Atlas weatherometer located at Natick Laboratories, Natick, MA. Simulated radiant sunlight (Xenon light source) was continuously maintained on a 12-hour light - 12-hour dark cycle for the total experimental run of 300 hours, i.e., the approximate equivalent to six months of outdoor exposure in a temperate climate. Temperature was maintained at 100 F; humidity was maintained at 50% RH, throughout the 300-hour test. This procedure is essentially as described in the FTM 406, method 6024. Samples of the two coatings were subjected to this exposure test; optical properties before and after testing are shown in Table 6. Samples were examined at the conclusion of the testing for cracking, crazing, or bubbling of the coating. Both coatings developed extensive cracking; the cracking on the 130 Plex II sample was, by far, the

Table 6. ACCELERATED WEATHEROMETER TEST

Atlas Weathermeter Chamber; Exposure 300 hr at 100 F, 50% RH, 12-hr Irradiation Cycle

	%H	Total Light Transmission	Yellowness Index	Abrasion Resistance % Haze at 1000 Cycle, Felt and Abrader		Comments
				%H	%T	
Plex II 130	1.8	93.1	-2.1	5.6	89.5	Optically Clear
Plex II 129c	1.1	93.4	-2.2	3.5	91.0	
Post 300 hr Exposure						
Plex II 130	2.6	93.7	-1.5	6.4	86.4	Severe Crazing after Exposure
Plex II 129c	1.7	93.1	-1.9	4.0	91.6	Crazing

more extensive, while the 129c Plex II sample crazed in a loose "spider web" pattern. Felt pad abrasion tests were run on the exposed samples. The results are compared to test values for virgin materials in Table 6. No bubbling or delamination of either coating was noted; nor was there any significant change in the abrasion resistance of either coating.

DISCUSSION

It may be concluded, based on assessment of the results of both abrasion tests, that the coating systems provide a significant improvement in the abrasion resistance of polycarbonate and acrylic plastics. The relative improvement achieved by the addition of the coatings to various substrate plastics is generally comparable or superior to other coating systems evaluated in previous studies.¹ It is felt this coating system offers promise in windshield application if significant improvements can be obtained in environment resistance and if commercial availability is achieved. It is recommended that further work be carried out to optimize these coatings.

Marks' coatings, when tested dry with the pad abrader, are at least four times superior in abrasion resistance to Abcite-coated polycarbonate (see Reference 1). The coating appears to be susceptible to ultraviolet and humidity conditions. Testing with a simulated and accelerated outdoor regime produces extensive and visually distracting microcracking; however, it does not appear to seriously affect abrasion resistance. It may be assumed that UV radiation was the primary factor producing the observed cracking. A possible mechanism would be the UV radiation affecting main-chain breakage of the PVA matrix; stresses within the coating (possibly at sites of microparticle inclusions) would subsequently cause the cracking. Microcracking, in all samples tested, occurred only in the coating, with no change noted in any of the substrates. (This observation is based on a discussion with Dr. R. Sacher, AMMRC.)

Similar cracking was also observed on post-test inspection of samples exposed to UV radiation alone. This is similar to that produced by the weatherometer testing and, similarly, no significant loss of abrasion resistance was noted.

Humidity, however, appears to cause a decrease in intermolecular bond strength within the PVA matrix, resulting in the coatings becoming softer and significantly less abrasion resistant. This was demonstrated by the abrasion test carried out on the "water soak" samples. The water soak samples were abrasion tested immediately on removal from the water bath (i.e., the PVA was infiltrated by water) resulting in about twice the haze development normally encountered by this test. The presence of water either during testing (windshield wiper test) or immediately preceding it (water soak test) was found to adversely affect the abrasion resistance of the 129c coating when applied to polycarbonate; this was witnessed by the 130 coating on polycarbonate giving superior abrasion resistance only in those two tests involving water while yielding inferior performance to the 129c coating with the remaining test methods. A possible explanation for this reversal of coating performance is the greater hydrophilicity of the polycarbonate over acrylic plastic. This would result in poorer surface adhesion with a PVA coating to the hydroscopic surface than to the relatively

"drier" acrylic surface. For all dry abrasion tests, surface adhesion becomes less influential in affecting coated performance; this results in the coating having the greater particulate material (130) giving the superior abrasion resistance.

Solvent testing of the coated plastic materials produced crazing on several of the coated polycarbonate samples; it was assumed that this crazing was due to solvent reaction with the polycarbonate plastic occurring through voids in the coating. This is the most logical explanation for the crazing reaction as PVA is soluble only in water or water and phenol. The particulate filler in the coatings is insoluble and unaffected by any of the organic solvents used in this test; therefore, the only reaction possible is between the test solvent and the substrate plastic materials.

Insufficient stretched acrylic materials were provided for inclusion in all the test methods. The only tests performed on this material were those which were judged most useful in assessing the performance of the coated plastic.

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